Notebook, N-09-07-13

This notebook contains dated entries from 1909, 1914, and 1915. All entries are by Edison. The book consists of notes and drawings regarding tests of various chemical compounds for use in phonograph records. The earliest entries indicate that the experiments probably applied to both cylinder and disc records. Inserted into the book is one loose page by an unidentified experimenter pertaining to the use of benzene and hydrochloric acid. The front cover is labeled "Disc Records 1909." The pages are unnumbered. Approximately 50 pages have been used.

July 13 1909

Experiments with Internal gwa 40 lb pressure

1 of Internal 3 of shellac or measuring amount of disturbances to see. we can find something that will make Compound Come free from mould Gelatin to get moist

Margin: — using a knife blade 1/2 finger nails measurement — Rag chips away from stick 1/4 of an inch of Gutta

No 1 Acetaminiche 1 2 3 4,

all stick to stick knife cracks off 1st. 1/8th — No 4 tough pliably — asphalum hard so can bend a little —

Phenacetin Stix only 1/8 chips Zoff —

Di chloro Gengal Green 500 gm

3 Expt 21 large Gelatin —

Make Milk Lumpie — Expt quite Steady

1st Gullon tougher — Chips 1/8 — after 1/2 hour 2nd 3rd + 4 Gullon pops up flexible —

4 Resorcin Chips 1/8th of area of Bullon

5 Benzophenol - makes it very Lumpy - Especially 3 & 4th Bullon - Enjoy flow 1/8 chip out last Bullon flexible -

6 = Fluorene
3rd & 4th Bullon mix
Very lumpy -

7 Carvanba - makes it about same thick - don't mix perfect - by stirring OK Emulsion

Fwd Sup now all he wants is something to make Tetrachlorap Shellac Compound more limpid at same temp so he can centrifuge it = Says Dinitrobenzol is OK but wants something cheaper as Dinitro has to be very pure to work —
Stearic makes it more limpid —

Benzophenol is OK $4.00 lb

Exalgin: very limpid. very orange grade —
The one I used considerable in flex — where used only little it OK — This was fairly limpid

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Phenylacetamide — No change in limpid —

Monomethylamp
limpid but too flex

Chlorinated Solvent Naphtha
wash Ethyl alcohol
Makes it Lumpy - acts
just same as Dinitrobenzol

D. Ettain new big lot of Celluloid

Dinitronaphtalene pure - very hard brittle
Cut 3 buttons - good Celyelozer -
4th button after 1/2 hour not
Crystal stix dried ~~but only~~
~~[illegible] although buttons~~
~~[illegible] brittle~~ no softening
M.P. is rather high - liquid is
black in all shapes - Turns
dirty white color crystals
Its a poor dei - Curious
Q 2.25 lb -

Very shiny surface on
Crystals buttons shows
glassy surface of dish -

Feby 12 1914.

Nickel flake mix removed from old returned cells - giving less than 100 dump
A4 —

Put in following Concentrate
Saturated Solution Cold
75 Fahr - 3 days -

Followed & disintegrated so flake & Ni can be separated

Potassium Sulfocyanate - possibly goes on Ni —

Sequette Salt when washed Ni goes Cadmium & Potassium Thorn fellow NG —

Potassium Sodium Tartrate & disolves some Ni — Works ok for disintegrating —

Potassium Citrate, disintegrated

Greenly is Equally with
disintegrating by using
Glycerin & KOH.
Spilling proper.
Consul proportions
this works fine —

Potassium Hyposulphite
Dissolves

Pot Bromide — Dissolves

Pot Chlorate " good
Nothing in Solution —

K Chromate No action
Na Nitrite very little disintegration Colors
liquid green — poly dissolves some

Na Nitrate No action
Na Sulphate "
Na Borate "
Na Chloride "

Up to this point K Chlorate is best,

Experiments Sept 20 1915
on Crude Phenol —

Treated with 1/2 & 1/2 H_2SO_4 & water — Separated & Evap'd down. The H_2SO_4 bal, turned same color as phenol — & gave considerable white crystals — These crystals only little soluble in water

Ditto Could HCl — gave a very large amount white crystals 6 or 8 times as much as H_2SO_4 & nearly all sol in water —

Appears to be some sulphate
asch Br — throws down 1/3 of the stuff — its probably a mixture —

Phosphoric acid Neg —

10% Conc HCl — filtered & Evap

50 cc phenol 12 cc Bi Sulphite
Saturated solution
Only leaves Bisulphate a very
little mush — looks good —

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50 cc phenol treated great
Excess Conc HCl — OK
only little fluid mush

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50 cc phenol a little dead
hydrox — followed — NG
lots mush

50 cc Phenol 10% HCl
Some muck-dry —
Wants more HCl —

50 cc phenol
10 gms cold fused
Bisulphate Na (fillers)
Lots Bisulphate dissolving
& some muck - Not quad

Another phenol
treated HCl - OK

50cc phenol
12 cc 1/2 + 1/2 H_2SO_4
Ok about same as HCl.
OK - Lumped small

50cc 2 1/2 cc HCl - Cone
Considerable muck
Shows enormous quantity
Salt dissolved in
phenol —

50cc phenol - glacial
phos acid - Lots muck
+ ~~Lots~~ sublimate
white crystals - what
are they —

Added Excess Conc HCl to phenol in 100 cc glass after 1/2 hour -

6 cc Solid Salt	7.19%
16 cc HCl —	19.51%
60 cc phenol	73.17

Residue of HCl & H_2SO_4 after driving off phenol & OK all dissolved in Acetone & mixture shaken & Acetone — NaCl salt, left

Solid Oxalic acid NG

Ignited Copper Sulphate - stirred in - NG

50 cc Rh .20 cc Conc HCl -
Very good - very little limpid residue -

Passed HCl gas thru 50 cc
Drops out large granular salts - Very little gas -

Experiments on
Paranitrophenylacetamide.

I took 100 grms of our 1.4 mixed acid & 26% Nitric

Used 20 grams CP Acetanilid powered powder gradually say in 5 minutes all in - Sparingly any heat is produced although it dissolves easily -

I moved flask at times in ice water with floating ice but it was probably unnecessary

A Water Cooled steel tank is all that I necessary I think -

The reason why it dont heat up is in my opinion

due to slow nitration I left it (after alles was dissolved) for 8 hours found on return the cobalt had almost gone solid although it was cold when I left it than it was when found solid –

Possibly it is all nitrated if not more H_2SO_4 caused 6-2 hours –

I then shook it & poured it out in beaker as a mush of Crystals added a little more water Cold –

Then passed in our small Vac filter got nice Cake yellow Tinge after pressing it down broke Vac & tried Cooling down in ice Method but practically only a milkness & no appreciable amount was in acid –

I then pressed Cake down hard got all liquid possible out added small amount of

Damp Nitroacetanilid is fairly soluble in Kerosene boiling

Acetanilid is much more sol—

Cold water, ~~[illegible]~~ 1/3" over the 3" Calc.—
this went then added another
1/3 of water, this went then
finally a 3rd —

The solution that came then
only showed Opal when
Chilled —

Evidently this is a fine way
to make Nitroacetanilid —
When proportions are ascertained

In Expanding with reduction of
Nitroacetanilide I guessed proportions
& got some fine Paper —

I then made a larger batch
according to Dr Swetzers
proportions

to wit

100 g Nitroacetanilid
500 dito Kerosene 116° C BP
250 " Iron 60 mesh
200 " 50° Be Soda Lyes

This would not reduce at all

in first Experiment lye liquor
intense black, in 2nd Experiment

liquor not black + iron Ferric
only result was a flocculent
stuff by Chilling out,

Evidently Soda too great excess

Of 20 grams Acetanilid used in the
8 hours nitrating got 22 grams
of what appears to be
Nitroacetanilide

I now put this 22 grms in reflux tube flask & add 60 cms petroleum 116° 130° to dissolve it, will add more till all dissolved when boiling —

Cant make any of Dudgeons formula work, neither can Chandler — yet I got it by guessing

We distilled Crude Para with Vac using screw & cloth at pump end & 2 pots after still — nearly all came down solid in 1st pot — Most came off at 205°C finally heat temp ~~off~~ up

to 255 @ 260-

Got off 85% 15% left of residue contained scarcely a trace of Para — Malaria is fine OK

This is OK

We also tried using the reduction pot mud after it is neutralized with Soda, distilled it - This appears to be OK will try another distillation & get yield. This cuts out water evaporation.

Phonograph

The only resinate soluble in Alcohol that is available as a substitute for Shellac is Resinate of Magnesia - Can get it syrupy fluid -

It softens but does not melt, right on hot plate - (B) the alcohol dissolved & Evaporated stuff - It is brittle & may not give proper strength to Disc.

Note -

Neoschema

Dissolve the substances in fume Benzol - Then Chill down Benzol till it solidifies Then in Crystalizing will throw out the thing dissolved in it -

Chill from bottom or sides

Get locks stuff in - use another poorer solvent mixed with it

Paraundophenol

Crude base -

Dissolves a little in

Crude 116° C BP Kerosene Hot & chilled out = flocculent ~~[illegible]~~

Very Rolin Acetone

190 BP Kerosene dissolves more but it may be in the Kerosene itself when I chill it

flocculent stuff tested in Paraldiphenol - it will be an increased amount of [illegible]

Solvents for Acetylparaphenylendiamin -

Acetone good

Pyridin good

Formaldehyde Hac fuse

Amylalcohol some

Benzylalcohol good amylacetate [illegible]

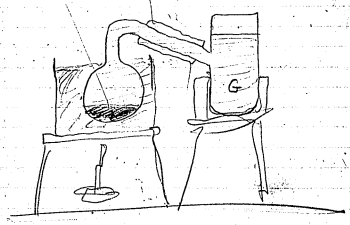
Benzol Methyl Tol Xyl - Carb Tetrach, poorer

Chloroform - Ether Benzine Turpentine -

Cyclohexan - Dont dissolve

Neo Scheme

X distill at say 220° Cent but decompose in air and vapor tension



The liquid used distills 325°C

X It should go over deposit in G

Alcohol Soluble Oils for Shellac in Calcutta to keep it from getting too brittle

Sassafras Oil Ok Dissolves
Origanum " "
Linseed N.G
Corn Oil N.G
Oil Cedar Wood Ok Dissolves
Safrol "
Rosin Oil fair.
Camphor Oil Ok Dissolves
Solvent Nap " "
Oil Cade - N.G " "
Heavy paraffin

over

~~H~~ Oel Benna NP
Ambrosia Oil — Ok Desalon
Castor Oil — Ok Desalon

Amedo Base —

Made by taking Reg Amedo liquor from filter press after Zinc is out, Neutralizing with NH till just acid + precipitate base —

Treated base with alcohol but till got out practically all —

Weight of Base 11.7 from
" of Zinc 9.5 "

It seems impossible to make Amedo Hydrochloride from the base made this way

Alcohol must decompose it or something wrong

Azo Dye Amido green
is reduced by Zinc dust
in Conc HCl — also by
NH4Cl —

Exhausting Para reduction
got dried & powdered Mass
with hot Alcohol —

75 grms powder
200 cc Al —

1st gave 25.250 grms Dry Para
phenylendiamin

2nd Exhaustion gave 1.150 gramm

3rd gave 98 milligramm

Residue dry 29.4 —

I will exhaust this water.

over

Took the mud after all is reduced
dried it in Vac dryer at 125° Fahr

Then powdered it thru 60 mesh

Then put the 75 grms into
boiling alcohol - stirred
for 5 minutes & poured it
into 3" Vac filter 2 papers
while stirrer going
Run through in 10 minutes
to bulked up mud - Took
this out & put it in 100
cc hot alcohol stirring
for 5 minutes thru it in
filter again — / When dry
Took it out again &
put in 50 cc Hot al -
& filtered again -

The alcohol got cold in
I want hot water
jacket around filter
to do good work -
a new one with this is
being made

29.4
26.3
3.1

Loss by acting on the 29.4 gram 3.1 grams

I have just powdered up some Crude Para mix right out of reduction pot & Vac Dried - found Enormous lot Coarse Iron not used.

After ~~[illegible]~~ powdering up & getting all Iron out then 60 mesh then powdering some of the iron found 644 grams of Metallic iron ~~not~~ going thru 60 mesh & 960 gram thru 60 mesh -

Experiments on Evap down
mother liquor of Amido ofles
Hydrochloric acid, its
first and — Chrysoidin calls
it Amido base

Took 1 lb paramido Chem
too much

Used 7540 cc water in 4
sections 175° Fahr —
Should say 5000 cc will do it
if Boiling carried in 5 sections
last lot can be used on
new lot —

116 grms ZnO dissolved in practically
free of any organic —
Ammonia Evaporating down
the water over

The amount of base from
the Evaporated water is

136
117
253
116 = Zn Reader
369

116 taken 450 grams
369
81

81 grams dry —

On ignition there is a white
residue which is probably
NaOH, no Zn —
This from prepared Gape
This must be got rid of

The Aryphenol Dye was
recovered got dimethylphenol from
Disclosure

1 Benzene Trace
2 Benzol Trace
3 D&N Alcohol good Solvent
4 Gasolene Trace
5 Camphor Oil fair
6 Kerosene Trace
7 Carbon Tetracl None
8 Pyridine fair to good
9 Chloroform Trace
10 Formaldehyde Trace
11 Amyl Alcohol fair
12 Benzaldehyde good Solvent
13 Fusel Oil fair
14 Petroleum Ether None
15 Acetate Amyl fairly sol
16 Glacial Acetic fairly sol
17 Wood Alcohol Sol Insoluble
18 Ammonia Some
19 Solvent Naphta Some

Amedothenol Hydrochlor

Solubility at 35° C. 19.0 grams in 100 cc

212° Fahr 77.0 grams in 100 cc

Boiling Alcohol
14 grams in 100 cc

Good Amedoplenal process –

After reduction filter Zinc metal off possibly – wash with boiling water –

Then neutralize so very faint acid.

Boil + filter – Chill out the Base, put water back with Cake from filter, Boil, then filter boiling – Chill liquid + get more base. Put Clear back on Cake + boil then Chill out base – finally when can get no base to Chill out. Put liquid on Cake + boil down to 1/2 bulk – filter boiling Chill out base. put liquor back on Cake + boil down to 1/4 of original bulk – filter boiling then Chill out

Evap small remainder of

liquor & save —

The various crops of Cane

show very little zinc or

other residue —

Now take base & add HCl &

small quantity water,

till clear but concentrated

because have proper HCl but

not too much — ~~HCl~~ HCl is to be

clear when cooling —

Cool & filter out crop of

Hydrochlorate — Then

to get a second crop

pass HCl cold through solution

you can get several

crops, finally evaporate

solution down to 1/2 &

Cool — pass HCl & you get

another crop on chilling

finally evap to dry & save

residue

The final Cake shows no smoke or change but keeps shape & turns white as Lunds Nell — it is black until burned —

From appearance of the Lunds Solution brought up by Christensen — it is 1st too dilute

2 I dont think the Dye is fully reduced —

When I evaporated down the small liquid residue about 25 cc I chilled out Dalt with base —

by doing this all the base can be got out clear without addition + dry salt can be added on by Alcohol

~~200 Dry Dye~~
~~200 cc 20% NaOH.~~
~~70 grams Zinc dust.~~
~~Checked — Gives more Zinc~~
~~Unsuccess,~~

OK =

570 Dye
450 Zn dust (ground process)
to 480,

500 lbs Solid Soda
add enough water to
make thick mush

Soap has 1st better Coke is
mix of Metallic Zinc
Zincate soda —
Dont go above 70 Cent,

to get the base, the necessary
degree is more machinery to
can be wrap to solid —
treated with alcohol

No Extra water is added
in this process at all

The crude base from the
200 cc weighs without ~~Solid~~
part while WET 18,950 gm
is now drying —

The base after drying
(from 200 cc) weighed 9,820 gram
This looks like full yield —

I treated the salt with alcohol but got only few milligrams - Probably unnecessary to treat salt or residue from final liquid
Weight from alcohol
45 mg, very impure I think

There is only 1½ lbs of pure for a 300 lb batch so not worth saving with trouble from alcohol manipulation

I took 50 grams of mother liquor amino residue powdered
Extracted in 200 cc water
Chilled ~~twice~~ Three times got 10.10 grms
good base out —
90% came out on first
Chilling — think that of

The powder is ground fine
150 mesh + 250 cc H_2O to 50
grams a slowly boiled
down to 175 cc that
35% fuls Chlor will take
out 95% of lime &
then evap to 75 cc all
will come out,

Amedo -
400 gals water
450 lbs NaOH solid
450 lbs Zinc dust
500 lbs Dyi or 1 batch

Benzidine -

Made batch in lab -
Took Casserole full
washed up Zinc far as
possible - filtered out
Soda highly diluted -
got pretty fair mechanical
separation of Zinc &
this is practical yet bulk
not easy -
The large bulk of Alkali sol
had a precip in Morning
filtered it off & found

Benzidine
it was Zinc — probably
decomposed from Zincate
by too great Excess of
Water — Must look
out for this —

The Hydroazobenzene I treated
with about 15% H_2SO_4
& got most of Zinc out
think Can get it all
out — but found some
Hydroazo in filtrate sol
showing it dissolves
somewhat in acid Solution
but not serious —

I ~~dere~~ powdered up the levigated Hydrozo put in 15% H_2SO_4 — & kept it warm 120° Fahr for ½ hour — stirring — The Hydrozo comes to top of liquid & needs stirring to get it in Contact with acid — The filtered solution when precip by NaOH showed lots Zinc — So this takes it out Easy & probably don't affect hydrozo —

Just tried filtering ~~Acc~~
Hydrazo crude from pot.
Diluted it with equal bulk
water, This weakens Alkali
so some Zincate decomp
It filters OK — Some of ZnO
runs thru dry cloth filter
but not thru same with
filter paper on top —
Well have to look out not
dilute alkali too much if
any but alcohol proceeds
as to be used — OK for Alcohol
if all soda washed out
so alcohol dont get loaded
with it —

Hydrocarbon is very soluble
Solvent Naph & Toluol, too sol in fact
to work out the chill and power
Kerosene works very good &
about 1/2 the solubility of Alcohol
Can bail the Kerosene which
floats above water & yet
all the Hydrogen comes out
beautifully white Each time
which is not the Case with
Alcohol –

It works out stuff right from
reduction pot filtered & washed
Line all in –

The only bug in the Kerosene adhering to the Crystals, ~~[illegible]~~ it seems impracticable to use Heat to dry as Hydro. will decompose to Azo

Could wash with Gasoline but this would be a dangerous Gasoline process

To get factory started will have to use Alcohol + then impurities afterwards —

1st Factory thing settled

Crude from reduction pot is to be diluted with equal bulk of water —

Run this through filter press + wash Cakes with water till practically free of NaOH, Cold water Can be used,

2nd Break up Cake Turn
on 1/4" screen in pans &
dry in Vacuum dryer at
not above 115 @ 120 28" Vac
It is best to get it as dry as
feasible to prevent Alcohol
getting full of water

3rd The Heat used to cause
Hydrazo to devolve should
be 15 to 20 deg below Boiling
point of Alcohol, I will
determine this exactly - this
is important, as heat
causes decomposition to
Azo & red color to Crystals
due to Azo -
Cold water 50° Fah or less

Well do for Cooking - but if lower temp can be obtained it will reduce the number of passes for dissolving & Chilling

The dried filter Cake from reduction pot, double bulk water, filtered to Cake & washed ~~we~~ with water - dried at 100° Fahr works OK with alcohol Even on boiling - The Azo from reduction tank shows up not reduced - it dissolves Even in alcohol than Hydrazo & think in

any Event Exhaustion with
Alcohol can take place just
at the boil + Every Exhaustion
gives colder Azo —

After Exhausting a batch
+ filling all cans out
by chilling + then added
large bulk of water
this precipitated Hydrogen
out like a milk or it
ran thru filter bad,
Almost impossible to
get it all by pouring
several times back over
Cake —
This process of precipitating

by water is Rotten-ing

So far Kerosene works best,

The only defect is Kero sticking to crystals —

Alcohol will do —

From alcohol, ~~the~~ Hydrazo

wets hand in 3% HCl solution —

in 30% Sol HCl wets perfect,

The 30% in Couple hours

70 cc ~~dissolved~~ clumps 1½ grams

Hydrotropo - very little dissolved
I diluted it greatly with
Water & neutralized it to
blue to fumes with
Milk lime - got quite a
Cake washed it 2 or 3
times on filter with
Water & dried it.

I found by chilling the
Water after filtering
Crystals of Benzoine
form but back on filter
very little - the Benzoin
is not slightly in water
we should chill solution
before filtering & use

Smallest quantity of water —

Think can use 30% HCl — & Add large quantity of Hydrazo & then only little water. It gets half-better cool at,

Note that when I use Kerosene, it gets very red & when batch of hydrazo is entirely exhausted & all chilled out Red is very Red showing out

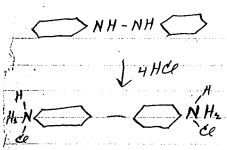
Reduction is very faulty &
some quicksilver is made
very soft in Ammonium chlor
Alcohol.

H_2 was 20% Kalth & hot Coals
I worked on was very warm
then attacked the Alcohol I
think -

Should be cold alcohol
put in & then Hydrogen -
I wonder if Alcohol is
really necessary -

[ITEM FOUND IN BOOK]

Hydrochloric Acid required for Conversion of Hydrazo-benzene into Benzidine Hydrochloride.



Hydrazo-benzene $C_6H_5 \cdot NH \cdot NH \cdot C_6H_5$

Benzidine Hydrochloride.

$4HCl = 146.0\,g$

$C_6H_5 \cdot NH \cdot NH \cdot C_6H_5 = 184\,g$

$146\,g\ HCl \Rightarrow 184\,g$ Hydrazobenzene.

$146 \times \frac{100}{184} = 79.5\,g\ HCl$ for 100 g Hydrazo-benzene.

$\frac{79.5}{1.19 \times .372} = 179.5\,cc$ of HCl (sp gr 1.19) for 100 g Hydrazo-benzene.

10/20 R.E.B.